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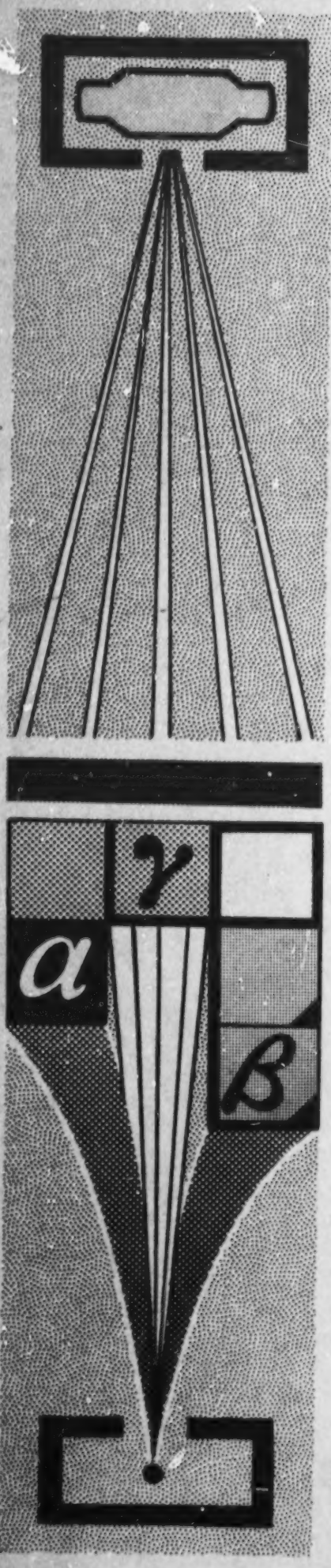

A SUMMARY REPORT on X-RAY DIFFRACTION EQUIPMENT

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A SUMMARY REPORT on X-RAY DIFFRACTION EQUIPMENT

by

Richard F. Boggs and Thomas M. Moore

INDUSTRIAL RADIATION UNIT
RADIOACTIVE MATERIALS SECTION
MEDICAL and OCCUPATIONAL RADIATION PROGRAM

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U. S. DEPARTMENT OF HEALTH, EDUCATION, AND WELFARE
Public Health Service

Bureau of Disease Prevention and Environmental Control
National Center for Radiological Health
Rockville, Maryland 20852

A SUMMARY REPORT ON X-RAY DIFFRACTION EQUIPMENT

Introduction

This report has been developed to review certain information that has been compiled by the National Center for Radiological Health. In an attempt to familiarize radiation safety personnel with the potential hazards in the use of x-ray diffraction units, a brief description of the fundamentals of x-ray diffraction, a review of the associated hazards, and suggested methods for proper evaluation of the x radiation produced is provided.

During the past five years, nearly 8,000 low voltage (less than 60 kV) industrial x-ray tubes have been sold in this country.(1) It is estimated that up to 80% of these tubes are being used in x-ray diffraction units. Although 3,000 - 5,000 such units are being used for a wide variety of analytical procedures, most operators and radiation safety personnel have a very limited knowledge of the hazards associated with the operation of x-ray diffraction units. As a result, radiation survey procedures have often been inadequate and potentially serious hazards from low energy x radiation have gone undetected.

Theory and Operation of Equipment

In 1912, M. von Laue developed an analytical procedure using x rays which could replace many wet chemistry procedures. He noted that the atoms in crystalline materials are arranged in a repeating

symmetrical three dimensional pattern, and that the spacing between the planes of the atoms approximates the wavelengths of x-rays.

The electrons associated with the atoms of a crystal scatter, or diffract, the incident x rays in a fixed manner. If a thin beam of x rays is directed into a crystalline substance, the emerging diffracted rays may be used to record a pattern on film. The pattern formed by these diffracted rays is characteristic of the crystalline structure of the specimen. It is therefore possible to use x-ray diffraction for applications such as identification of crystalline material or changes in crystalline structure.

Many methods have been devised for recording the diffraction pattern on film. Figure 1 shows the Laue method in which the primary beam passes through a single crystal. A flat film is used to record those rays which are diffracted in the forward direction. The primary beam should be stopped at the film with a small piece of lead, to prevent unnecessary fogging of the film. Figure 2 shows the back reflection method, which is used when the specimen is toc thick for adequate penetration of the x-ray beam. In this method, the rays diffracted back toward the x-ray source are recorded on a film. Figure 3 shows a method developed by Debye-Scherrer-Hull, in which a powdered specimen is used. The sample is placed in a fine glass capillary or on a material transparent to x rays. The diffract-ec rays are recorded on a strip of film which is placed around the specimen.

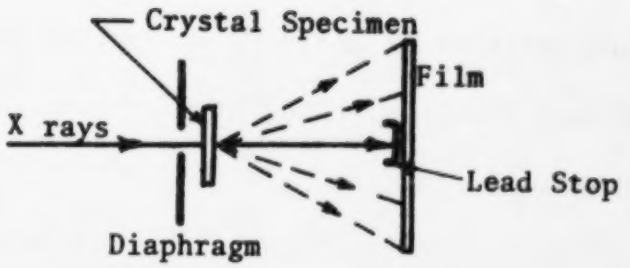

Fig. 1. Laue Method - Heterogeneous x rays and stationary crystal.

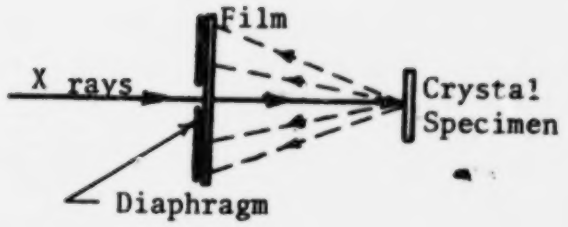

Fig. 2. Back reflection method.

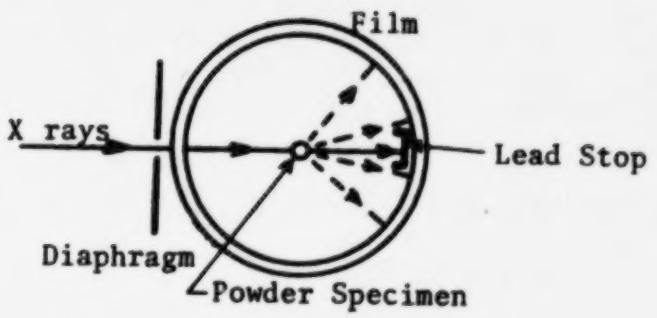

Fig. 3. Debye-Scherrer-Hull (powder) method.

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The diffraction pattern on the film can be analyzed by comparison with known standard patterns or analyzed by application of Bragg's Law.*

An alternate, non-film, method of recording the diffracted x rays provides faster, more accurate results. In this method, called diffractometry, a detector is rotated in an arc around the specimen; the detected impulses are amplified and recorded as peaks on a paper strip chart. The recorded peaks, which correspond to diffraction lines on a film, may then be used for both qualitative and quantitative analysis of the specimen.

X-Ray Diffraction Tubes

X-ray diffraction studies require well regulated, low energy x rays. These are obtained from x-ray tubes which operate at a maximum of about 50 kV. Maximum tube currents are about 45 mA when operating at lower voltages. These tubes have a very thin window which is usually made of beryllium.

The thin windows allow the passage of a high flux of "soft" x rays, resulting in exposure rates of up to 3×10^6 R/minute at the tube housing port.[2][3][4] The tubes usually have three or four exit ports, all of which may be used simultaneously.

A wide variety of target materials is used in x-ray diffraction tubes. The target material is chosen according to its emission spectra. The spectrum requirements are dependent upon the specimen which is to be analyzed.

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*The description of x-ray scattering phenomena has been reduced to the following equation for reflection angle; this equation is known as Bragg's Law.

$$n\lambda = 2d \sin \theta$$

where n = order number
λ = wavelength of incident x rays
d = distance between planes of atoms
θ = angle between incident x rays and atomic planes

Table 1 lists the commonly used targets along with their characteristic K_α and K_β wavelengths. Copper is the most popular target material; however, the others are readily available for special requirements. Also included in Table 1 is the proper beta filter for use with each target. The beta filters remove the characteristic K_β wavelength, producing a nearly monoenergetic x-ray beam.

TABLE 1

Characteristic Spectra and Beta Filters for Common Diffraction Tube Targets

Target	Characteristic Wavelength				Beta Filter
	K_α		K_β		
	Angstrom	keV	Angstrom	keV	
Copper	1.54	8.01	1.39	8.92	Nickel
Molybdenum	0.709	17.5	0.632	19.6	Zirconium
Iron	1.94	6.41	1.76	7.07	Manganese
Tungsten	1.48*	8.40	1.28**	9.67	--
Chromium	2.29	5.42	2.08	5.94	Vanadium
Nickel	1.66	7.48	1.50	8.27	Cobalt
Cobalt	1.79	6.94	1.62	7.65	Iron
Silver	0.559	22.2	0.497	25.2	Palladium

*L_α

**L_β

Conversion Formula: $E\ (ev) = \frac{1.242 \times 10^4}{\lambda\ (\text{Å})}$

Radiation Hazards

As stated above, an x-ray diffraction unit may simultaneously radiate four very small beams of low effective energies. The beams are frequently in a horizontal plane, about four or five feet above the floor. These machines may be operated continuously for periods ranging from a few minutes up to many hours.

Personnel film badges usually fail to indicate this exposure because 1) the beam is extremely narrow and 2) the film badge is normally not worn at a height comparable to that of the x-ray beam. Many personnel overexposures from x-ray diffraction units, resulting in severe damage to fingers and eyes, have been reported. [2][3][5]

These are some of the most common causes of unnecessary radiation exposures from x-ray diffraction equipment:

1. Placement of fingers in the primary beam while changing samples because of failure to either block primary beam or stop x-ray production.
2. Visual beam alignment without using a leaded glass shield. This may result in the primary beam directly striking the eye for prolonged periods.
3. Failure to realize that x-ray beams are being emitted from exit ports other than the one of immediate concern.
4. Unattended operation of the unit in an uncontrolled area. Personnel who are unaware that the unit is operating may stand near or pass through areas of high scatter radiation.

5. Alteration or removal of shielding in order to perform a specialized analysis.
6. Installation or modification of equipment by manufacturer's representatives who perform final adjustments, etc. with the unit partially disassembled.

Survey Procedures and Safety Standards

Some of the basic points that must be considered in performing an adequate radiation survey are covered below.

The surveyor should discuss the normal operating procedures with the operator so that the usual machine settings may be duplicated during the survey. This discussion will also enable the surveyor to make suggestions for improving procedures such as sample changes and beam alignment.

In measuring the primary beam output (which is not usually necessary), two major factors should be considered:

1. The primary beam is extremely narrow and covers only a small portion of the sensitive volume of most monitoring chambers. Fluorescent paper may be useful in determining field size or in searching for "pinhole" leaks around the housing.
2. The effective x-ray energy is very low (usually about 5-15 keV) and requires the use of specially calibrated survey instruments. Most standard survey instruments are calibrated for use with higher energies and will not give accurate readings if used to monitor soft x rays.

It is desirable to have a large warning light or sign, which turns on automatically when the x-ray tube is activated. This signal should be positioned so that it will be immediately visible to all persons in the area of the unit. The small "x-ray" light on the control panel of many units is not a satisfactory substitute.

The surveyor should make certain that the console contains an easily read statement warning that the equipment emits x rays when energized. In addition, a standard radiation symbol should appear on or near the operating console.

No standards group in the U.S. has yet established guidelines specifically for x-ray diffraction equipment, although certain safety criteria have been developed.(6)

National Bureau of Standards Handbook 93, entitled "Safety Standard for Non-Medical X-Ray and Sealed Gamma-Ray Sources, Part I. General" pertains, in part, to diffraction apparatus; however, a complete set of standards is not included. Other NBS handbooks allude to x-ray diffraction equipment.

Following is a list of known manufacturers of x-ray diffraction units, tubes, and/or associated equipment:

Picker X-Ray Corporation

Philips Electronic Instruments (Norelco)

General Electric Company

Siemens America, Inc.

The Machlett Laboratories, Inc.

Dunlee Corporation

Elliot Electronics Tubes Limited

Electronics and Alloys, Inc.

Polaroid Corporation

Reuter-Stokes Electronic Components, Inc.

LND, Inc.

Hamner Electronics Company, Inc.

X-Ray Spectrographic Equipment

This report has been confined to a discussion of x-ray diffraction equipment. However, another major type of analytical x-ray equipment, the x-ray spectrographic or fluorescence unit, has achieved comparable popularity. These units generally operate at slightly higher voltages than the diffraction units, but produce a similar x-ray spectrum. The two types of equipment are quite similar in operation and, in general, safety criteria are the same. X-ray spectrographic equipment is potentially more hazardous, because it is likely to undergo frequent modification to suit changing requirements.

References

(1) Based on information received from the National Electrical Manufacturers Association.

(2) McBride, W. W., and Lieben, Jan, Radiodermatitis from Diffraction Apparatus, Archives of Environmental Health, 4:191-195 (Feb. 1962).

(3) McLaughlin, J. E., and Blatz, Hanson, Potential Radiation Hazards in the Use of X-Ray Diffraction Equipment, American Industrial Hygiene Association Quarterly, 16:2 (June 1955).

(4) Radiation Hazards Associated with X-Ray Diffraction Techniques, Acta Cryst., 16:324-28 (1963).

(5) Howley, J. R., and Robbins, C., Radiation Hazards from X-Ray Diffraction Equipment, Radiological Health Data and Reports, 8:245-9 (May 1967).

(6) Beu, K. E., American Crystallographic Association Apparatus and Standards Committee for 1962. Safety Considerations in the Design of X-Ray Tube and Collimator Couplings on X-Ray Diffraction Equipment.